

Report SM-49105-Q2

STRESS CORROSION CRACKING OF TITANIUM ALLOYS AT  
AMBIENT TEMPERATURE IN AQUEOUS SOLUTIONS

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## FOREWORD

This report was prepared by the Astropower Laboratory, Douglas Aircraft Company, Inc., under NASA Contract NAS 7-488. The work is administered by Chief, Research SRT NASA Headquarters, Code RRM, with Mr. W. Raring as Project Scientist. This report was prepared by Dr. C. B. Gilpin and Dr. T. L. Mackay under the direction of Dr. N. A. Tiner. Mr. R. G. Ingersoll and Mr. W. A. Cannon have greatly contributed to the performance of this work.

## ABSTRACT

Specimens of Ti-5Al-2.5Sn, Ti-6Al-4V, Ti-8Al-1Mo-1V and Ti-13V-11Cr-3Al were prepared for stress corrosion tests (single edge notch specimens), transmission electron microscopy, and electron microautoradiography.

Stress corrosion tests in 3% sea water were initiated.  $K_{IC}$  values for Ti-8Al-1Mo-1V in argon averaged 44.2 ksi $\sqrt{\text{in.}}$ , whereas  $K_{IC}$  values in sea water were between 18.5 and 28.0 ksi $\sqrt{\text{in.}}$  and in distilled water between 30.4 and 36.6.

Electron microautoradiographic studies of the distribution of hydrogen in titanium alloys were initiated. Radioactive hydrogen (tritium) was introduced by cathodic charging and by gas adsorption at elevated temperature. Preliminary autoradiographs showed tritium introduced by cathodic charging to be distributed evenly throughout the lattice in the Ti-5Al-2.5Sn alloy. In the Ti-6Al-4V alloy no tritium was observed in the beta phase. At low charging current densities, tritium was evenly distributed in the alpha lattice, whereas at higher current densities tritium appeared to concentrate in alpha-beta boundaries and alpha-alpha boundaries. No tritium was observed in the beta alloy Ti-13V-11Cr-3Al.

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## 1.0 INTRODUCTION

Titanium alloys are found susceptible to stress corrosion cracking at ambient temperature in aqueous solutions. This may be a serious problem in aircraft and space vehicle applications, and very little is yet known about the nature of stress corrosion.

The objectives of this program are to conduct a detailed industrial and government agency survey and to carry out an experimental program to define the microprocesses in stress corrosion failure of different titanium alloys at ambient temperature in aqueous salt solutions. The experimental methods used for these evaluations include (1) examination of stress corrosion cracked surfaces by microfractography, (2) determination of preferential attack by transmission electron microscopy, (3) determination of the role of hydrogen, if any, by microautoradiography, and (4) evaluation of possible contribution of electrochemical reactions by potentiostatic measurements. The techniques of using these experimental methods were developed previously in other programs conducted by Astropower Laboratory on stress corrosion cracking of high strength steels. (1)

The work reported during this quarterly period deals with a preliminary evaluation of  $K_{IC}$  values for titanium alloys in a neutral argon atmosphere and aqueous solutions to evaluate their susceptibility to stress corrosion cracking. Microautoradiography studies were initiated to determine the preferential retention of hydrogen in the various phases present in titanium alloys.

## 2.0 EXPERIMENTAL EVALUATIONS

### 2.1 Selected Alloy Sample Characteristics

The following alloys were selected<sup>(2)</sup> for detailed investigation of the mechanism of stress corrosion cracking.

1. Ti-5Al-2.5Sn (an alpha alloy)
2. Ti-6Al-4V (an alpha-beta alloy)
3. Ti-8Al-1Mo-1V (a super alpha alloy)
4. Ti-13V-11Cr-3Al (a beta alloy)

Sheet materials of Ti-5Al-2.5Sn, Ti-6Al-4V and Ti-8Al-1Mo-1V were obtained from Titanium Metals Corp.; the thickness of Ti-5Al-2.5Sn and Ti-6Al-4V alloys was 0.075 inch while the thickness of the Ti-8Al-1Mo-1V alloy was 0.109 inch. A sheet of Ti-13V-11Cr-3Al alloy, 0.075 inch in thickness, was obtained from Crucible Steel Company. The chemical composition of the alloys as stated by manufacturer are shown in Table I. The standard heat treatment for each alloy is shown in Table II with mechanical properties as stated by the manufacturer.

The microstructure of each alloy is shown in Figure 1a, 1b, 1c, and 1d. Only the beta alloy, Ti-13V-11Cr-3Al showed a coarse grain structure. The microstructure of the two alpha-beta alloys, Ti-8Al-1Mo-1V and Ti-6Al-4V, exhibited a continuous alpha matrix.

A sheet of Ti-8Al-1Mo-1V alloy, 0.045 inch thickness, with a heat treatment described by Hatch, et al.,<sup>(3)</sup> to produce a discontinuous alpha in a beta matrix has been recently obtained from Titanium Metals Corp., and will be included in this program. The chemical and physical properties of this material will be included in the next report.

### 2.2 Stress Corrosion Tests of Alloy Samples

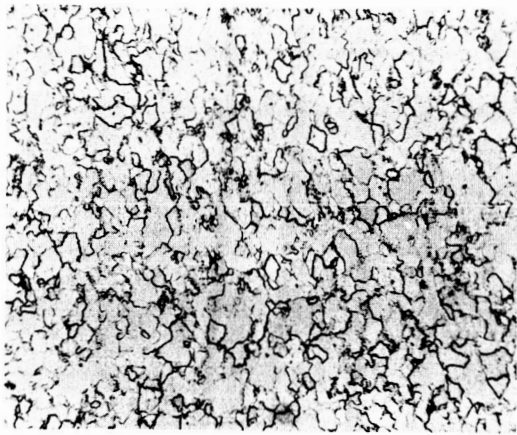
It is apparent from the literature survey<sup>(2)</sup> that the ambient temperature stress corrosion of titanium alloys is controlled with an initial crack propagation. Much work has been recently conducted on evaluating the crack extension force,  $G$  or  $K$ , in fracture toughness data<sup>(4)</sup>. It has been found that  $G_c$  or  $K_c$  (the value of  $G$  at the point of instability) is dependent on the specimen

TABLE I  
COMPOSITION OF TITANIUM ALLOYS

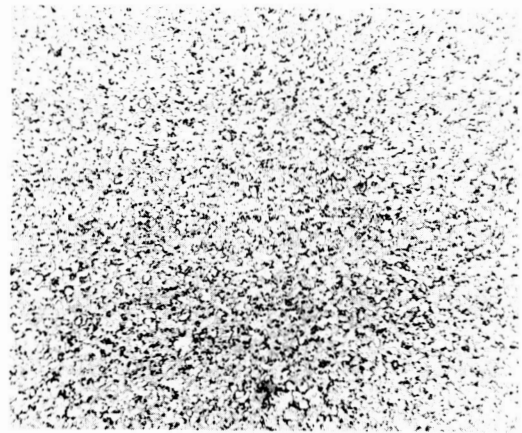
<u>Alloy</u>	<u>Al</u>	<u>Sn</u>	<u>V</u>	<u>Fe</u>	<u>Cr</u>	<u>C</u>	<u>O</u>	<u>N</u>	<u>H</u>
5Al-2.5Sn	5.0	2.4		0.27		0.25	0.10	0.015	0.007
6Al-4V	5.8		3.9	0.12		0.023	0.13	0.019	0.004
13V-11Cr-3Al	3.5		13.6	0.22	10.2	0.04		0.03	0.0154

TABLE II  
MECHANICAL PROPERTIES OF TITANIUM ALLOYS FOLLOWING  
STANDARD HEAT TREATMENTS

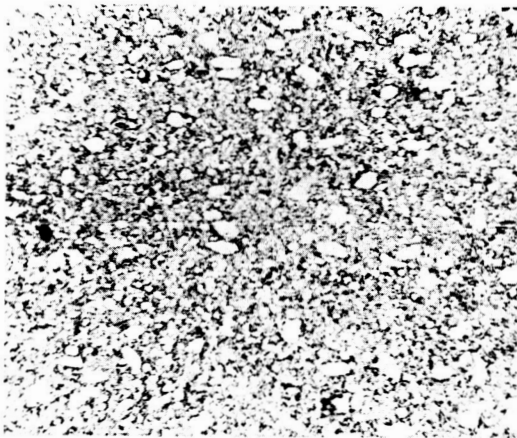
<u>Alloy</u>	<u>Heat Treatment</u>	<u>0.2% Yield Ksi</u>	<u>Ultimate Tensile Ksi</u>	<u>El %</u>
Ti-5Al-2.5Sn	Mill anneal (1325-1550°F, 10 min- 4 hr, air cool)	130.1	141.9	15.5
Ti-6Al-4V	Mill anneal (1300-1550°F, 1.8 hr slow cool to 1050, air cool)	138.0	144.5	13.0
Ti-8Al-1Mo-1V	Duplex anneal (1450, 8 hr furnace cool + 1450, 1/4 hr, air cool)	143.0	152.0	14.0
Ti-13V-11Cr- 3Al	Mill anneal (1400-1500°F, 1/4-1 hr, water quench)	140.4	143.2	23.4



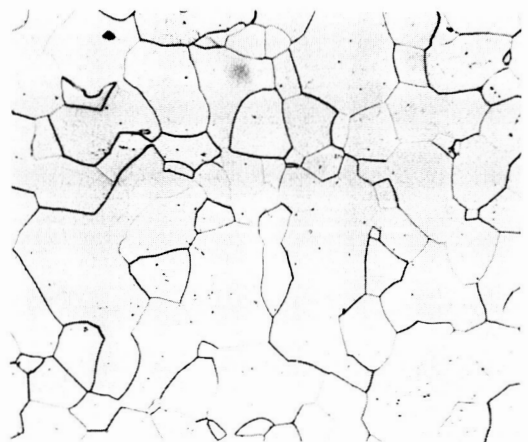
(a)



(b)



(c)



(d)

- a. Ti-5Al-2.5Sn
- b. Ti-6Al-4V
- c. Ti-8Al-1Mo-1V
- d. Ti-13V-11Cr-3Al

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Figure 1. Microstructure of Titanium Alloys, Mag. 200X

configuration. It has also been found that the plane strain fracture toughness,  $G_{Ic}$  or  $K_{Ic}$ , is independent of any specimen dimension and thus provides a material characteristic. From this work several specimen types suitable for  $K_{Ic}$  measurements have evolved.

The single-edge-notched plate configuration was selected for this program. Figures 2 and 3 show specimen configuration for the four alloys. This specimen configuration conforms to presently known requirements for plane strain fracture toughness measurements. Twenty-five specimens have been prepared for each alloy.

The corrosive solution is contained in a lucite box sealed to specimen with an epoxy resin to prevent leakage. The specimen is loaded in a Dillon tensile machine shown in Figure 4. The load is measured with a Baldwin-Lima-Hamilton SR-4 Load Cell which is capable of measuring the total load within four pounds.

Stress corrosion tests have been performed for the Ti-8Al-1Mo-1V alloy in 3% NaCl solution at ambient temperature and in distilled water. The load was increased in increments of 20 lb. starting at 1000 lb. The load was held for 5 minutes at each load level. The fatigue crack was observed with a stereo microscope and the time of initiation of crack extension was measured with a stopwatch. The stress intensity factor, at crack instability,  $K_c$ , was calculated using the following equation<sup>(4)</sup>:

$$K_c^2 = \left(\frac{P}{B}\right)^2 \frac{1}{W} \left[ 7.59\left(\frac{a}{W}\right) - 32\left(\frac{a}{W}\right)^2 + 117\left(\frac{a}{W}\right)^3 \right] / (1-\nu^2) \quad (1)$$

where

P = load

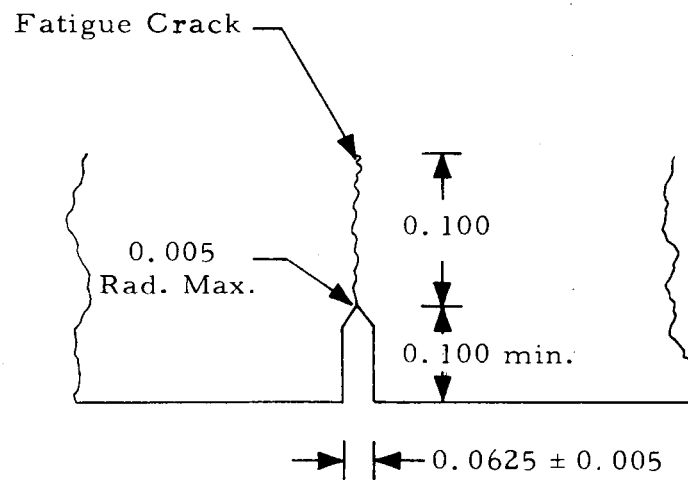
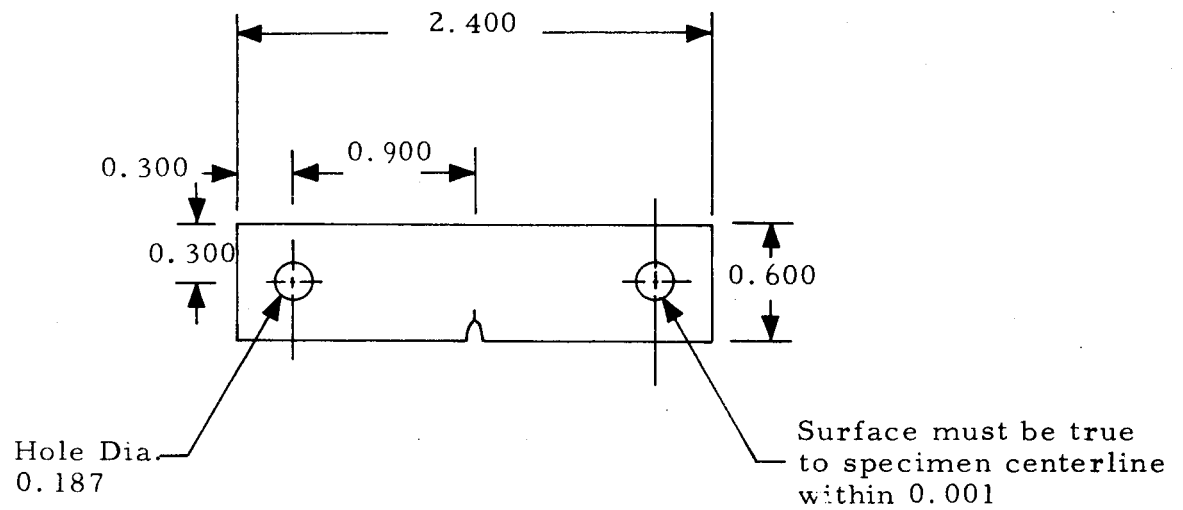
B = thickness

a = crack length

W = width of specimen

$\nu$  = Poissons ratio

The above procedure employs static loading, and it is not possible to measure the critical stress intensity factor,  $K_{Ic}$ , by this technique.



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Figure 2. Stress Corrosion Specimen Configuration For 0.075 in. Thick Titanium Alloys Ti-5Al-2.5Sn, Ti-6Al-4V, and Ti-13V-11Cr-3Al



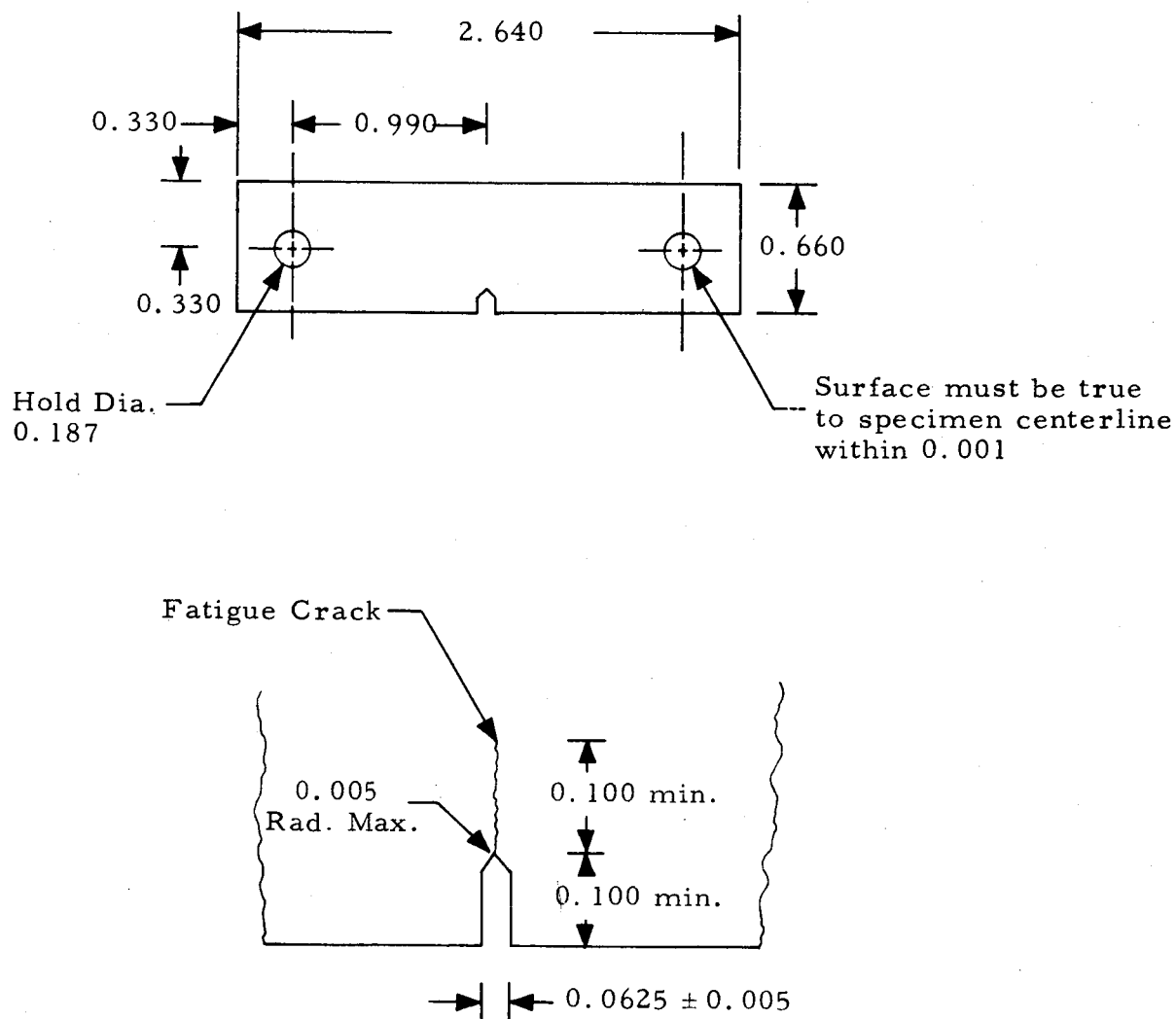
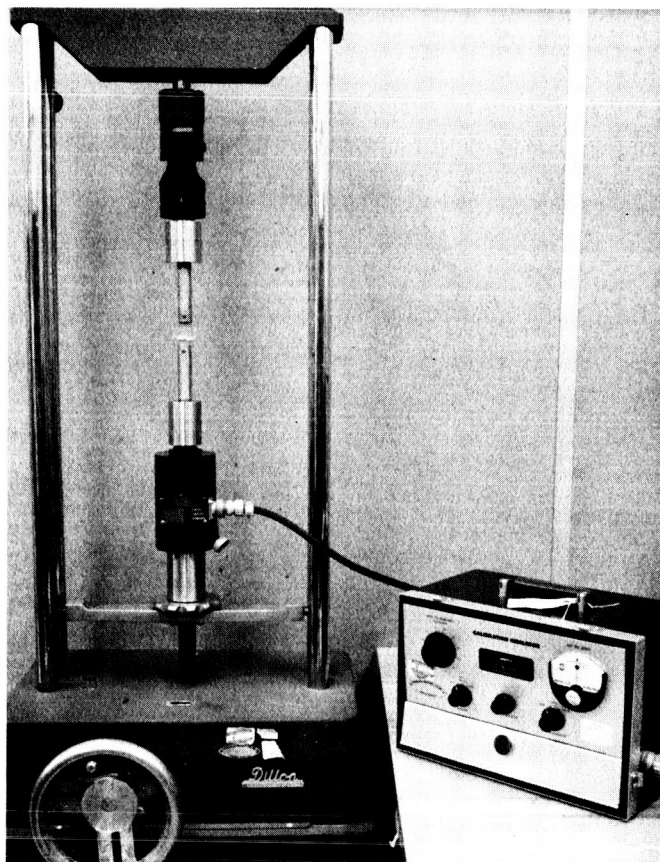


Figure 3. Stress Corrosion Specimen Configuration For  
0.109 in. Thick Titanium Alloy, Ti-8Al-1Mo-1V



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Figure 4. Stress Corrosion Apparatus

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The initial fatigue crack length,  $a$ , in Equation 1 was measured with a low power microscope following fracture of the specimen, and was used to calculate  $K_{IC}$ . Figure 5 shows a typical fatigue crack surface. The stress intensity factor,  $K_{IC}$ , to propagate a crack in argon, in a 3% salt solution and in distilled water for Ti-8Al-1Mo-1V is shown in Table III.

It was observed that there was a delayed time between application of load and crack propagation for sea water and distilled water stress corrosion cracking. Table IV shows the time for initial crack propagation and total time for failure.

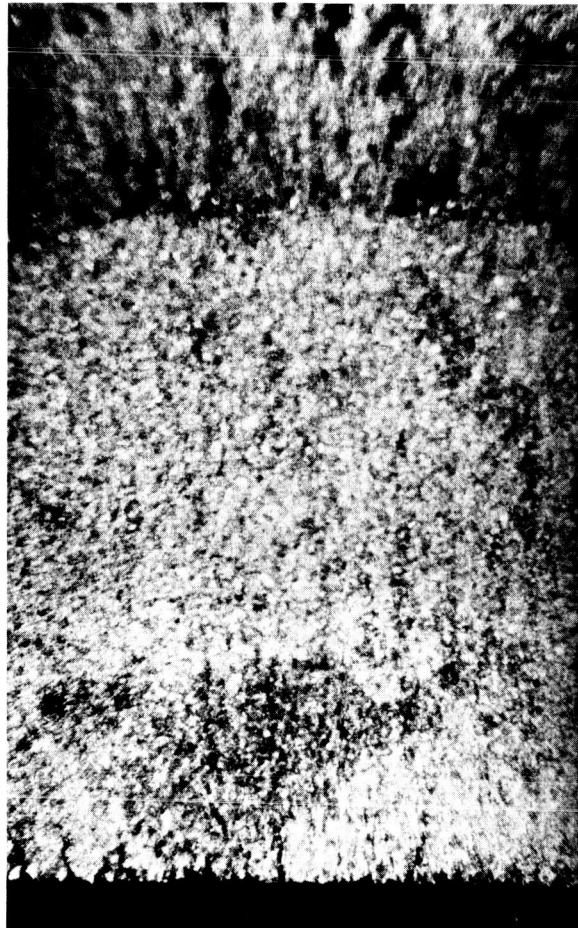
The reduction of stress intensity factor,  $K_{IC}$ , by 3% sodium chloride solution and by distilled water when compared with values obtained in argon shows that the duplex annealed Ti-8Al-1Mo-1V alloy is susceptible to stress corrosion cracking at ambient temperature in these environments. The sodium chloride environment lowered the stress intensity factor,  $K_{IC}$ , to a much lower value than distilled water. Using a 3% sodium chloride solution and distilled water, it will be possible to evaluate the role of chloride concentration and hydrogen diffusion on the mechanism of stress corrosion cracking of titanium alloys in aqueous environments. Electron fractograph studies will be made of the fractured surfaces in order to determine the mode of stress corrosion failure.

### 2.3 Study of Hydrogen Distribution in Alloy Samples

To study the distribution of hydrogen in various titanium alloys and its role in stress corrosion cracking, the radioactive hydrogen (tritium) was introduced into the titanium alloys by two methods; (1) cathodic charging and (2) gas adsorption at elevated temperature.

#### 2.3.1 Cathodic Charging

Tritium was cathodically charged into titanium alloys in order to determine the preferential segregation of hydrogen under these conditions. This was done to simulate conditions during corrosion of titanium in sea water in which hydrogen may possibly be charged at local cathodic sites. The procedure described was adopted in order to introduce fairly large quantities of tritium.



← Plane Strain Fracture

← End of Fatigue Fracture

← Fatigue Fracture

← Start of Fatigue Fracture

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Figure 5. Fracture Surface of Ti-8Al-1Mo-1V. Mag. 40X.

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TABLE III  
STRESS INTENSITY FACTOR,  $K_c$ , FOR Ti-8Al-1Mo-1V IN ARGON AND  
3% SALT SOLUTION AT AMBIENT TEMPERATURE

<u>Specimen No.</u>	<u>Environment</u>	<u><math>K_c</math> (Ksi<math>\sqrt{\text{in.}}</math>)</u>
1	Argon	43.7
2	Argon	44.8
3	3% Salt	23.0
4	3% Salt	18.5
5	3% Salt	25.2
6	3% Salt	28.0
7	Distilled water	36.6
8	Distilled water	30.4

TABLE IV  
TIME FOR CRACK PROPAGATION AND FAILURE OF Ti-8Al-1Mo-1V  
IN 3% SALT SOLUTION AND DISTILLED WATER

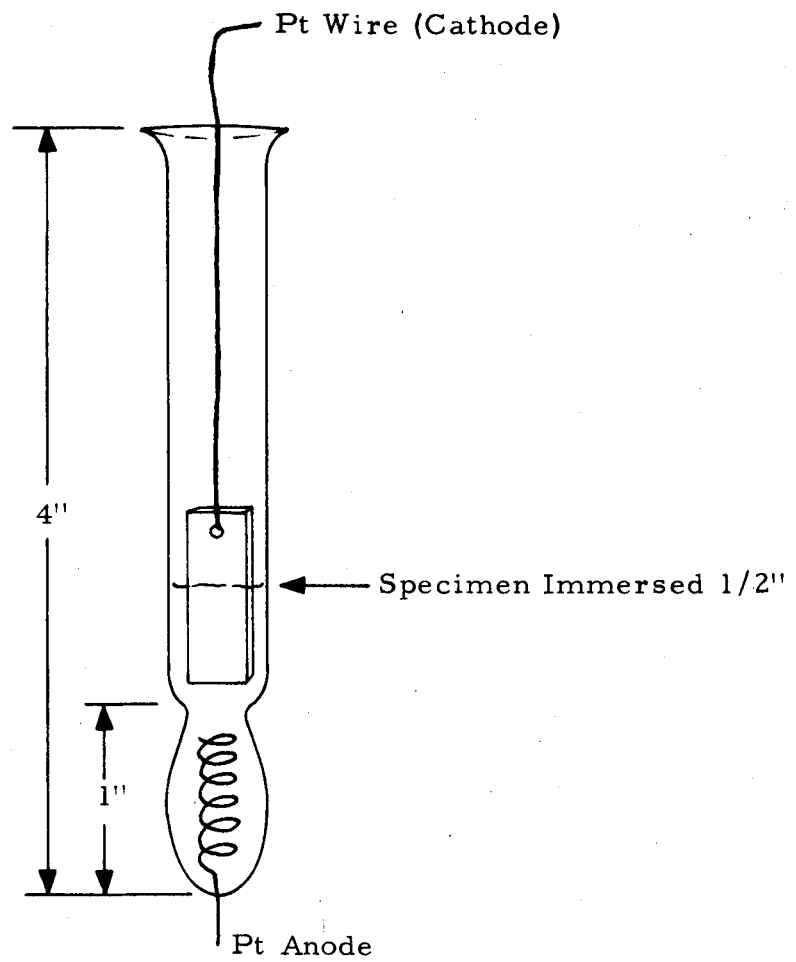
<u>Specimen</u>	<u><math>K_c</math></u>	<u>Crack Propagation (sec)</u>	<u>Failure (sec)</u>
3	23.0	—	60
4	18.5	40	82
5	25.2	60	110
6	28.0	25	38
7	36.6	70	132
8	30.4	70	125

The four alloys previously described were used for this experiment. Samples 1" x 1/4" thickness were metallographically polished. The final step in the polishing procedure was an etch polish using Krolls reagent as the etchant. Immediately after polishing, the specimens were cathodically charged in the cell shown schematically in Figure 6. The anode was platinum. The solution was 4%  $H_2SO_4$  made with tritiated water with a specific activity of 1c/gm. This solution has approximately one  $H^3$  atom for every 3400  $H^1$  atoms.

The specimens were immersed approximately 1/2" and charged at a current density of 80 ma/in<sup>2</sup> or 0.4 amps/in<sup>2</sup> for one hour. Specimens were then rinsed, dried, metallographically polished, etched, and examined in a light microscope. The electron microautoradiographs were prepared as follows:

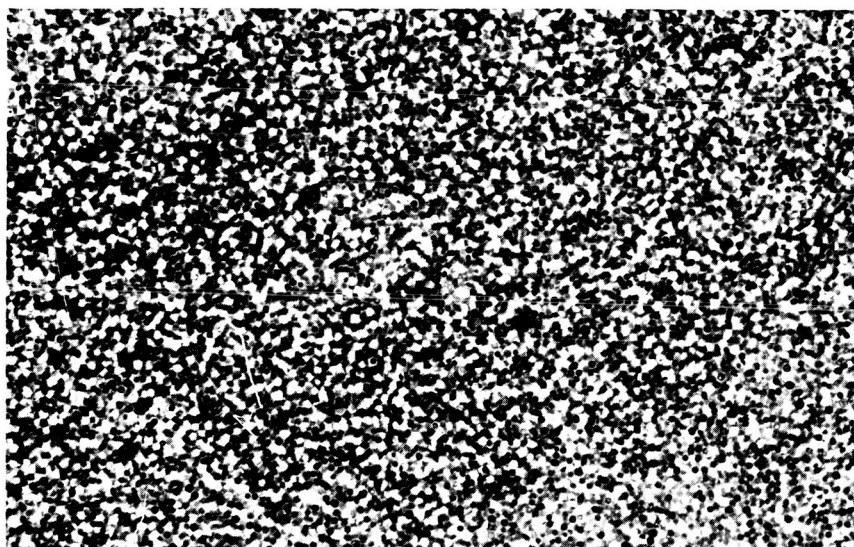
1. Ten angstroms of chromium were shadowed at 20 degrees. The chromium was placed on the specimen to act as a shadow for the direct carbon replica.
2. 150 angstroms of carbon were deposited onto the titanium for a direct replica of the metal surface.
3. A monolayer of Kodak NTE nuclear track emulsion was then placed on the surface. Figure 7 shows an emulsion film prepared on a carbon substrate at the same time.
4. The specimens were exposed for 11 days.
5. The emulsion was developed while on the specimen, and fixed.
6. The carbon replica and emulsion were removed with nitric acid and hydrofluoric acid.
7. The autoradiograph was rinsed, dried, and examined in the electron microscope.

Before describing results a brief description of the characteristics of tritium in titanium should be discussed. Tritium is a beta emitter with a half-life of 12.26 years. The beta particle has an energy of 0.018 MeV and thus has a maximum penetration in titanium of 1450 angstroms. Thus all tritium deeper in the metal than 1450 angstroms will not be recorded on the emulsion. Furthermore, since a beta particle may emit in any direction this means that the minimum resolution is 1450 angstroms, i. e., any silver filament observed on the radiograph must be within 1450 angstroms of the

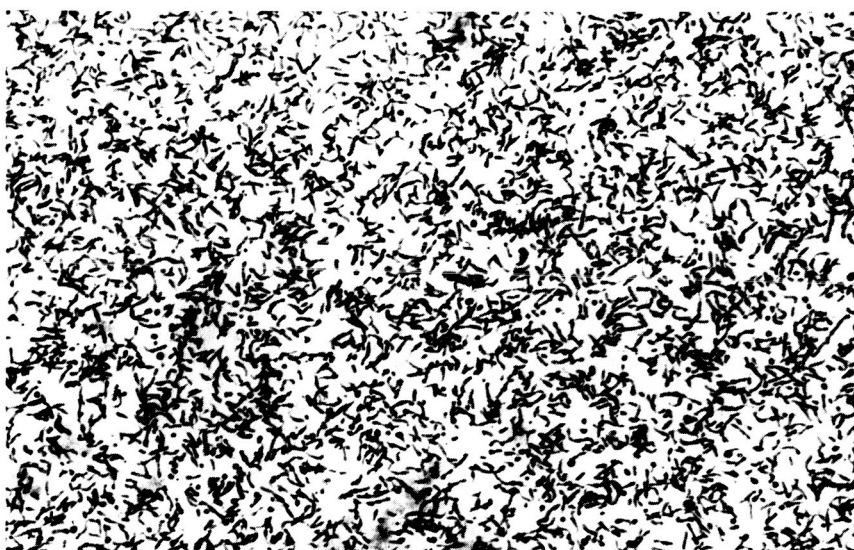


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Figure 6. Schematic Drawing of Cell for Cathodic Charging



(a)



(b)

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- a. As cast on carbon film. Round particles are AgBr. Mag. 17500X.
- b. Exposed to light and developed. Black particles are silver filaments. Mag. 26000X.

Figure 7. Autoradiographic Emulsion



actual location of the tritium atom that emitted the beta particle which caused that particular silver filament.

The emulsion was selected that had the minimum particle size, which for NTE is about 500 angstroms. The range of beta from tritium is 1000 Å in AgBr and thus a monolayer of AgBr does not stop all of the beta that reaches the emulsion. A thicker layer (i. e., more than a monolayer) can be used to stop all beta but the resolution would be decreased. Thus the autoradiographic method used for this experiment cannot be interpreted to calculate the concentration of tritium unless an emulsion efficiency is determined. This has not been done yet.

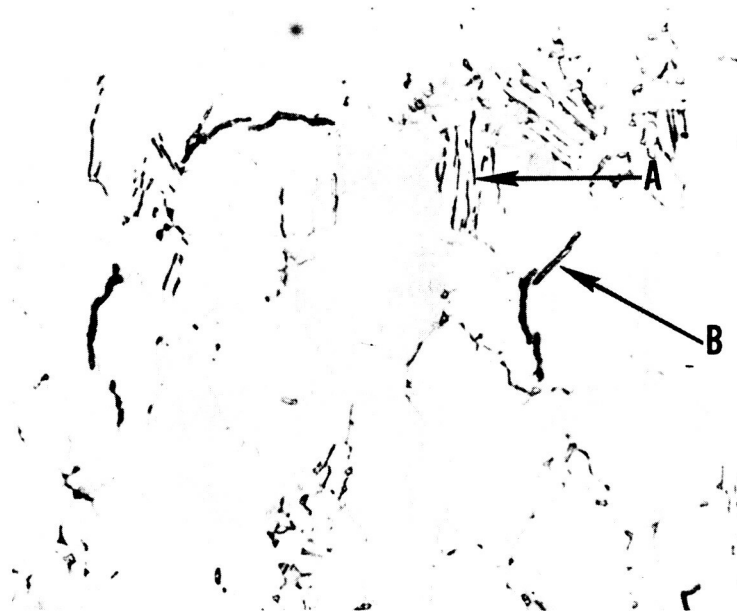
#### 2.3.1.1 Ti-5Al-2.5Sn Alloy

The Ti-5Al-2.5Sn alloy was cathodically charged at 80 ma/in<sup>2</sup>. After charging, hydrides were observed at the grain boundaries and within some of the grains as shown in Figure 8. Electron autoradiographs showed the tritium to be evenly distributed throughout the lattice, Figure 9. There was no apparent concentration at boundaries or even at hydride precipitates. This may mean that hydrides are forming with H<sup>1</sup> and not H<sup>3</sup> atoms.

#### 2.3.1.2 Ti-6Al-4V Alloy

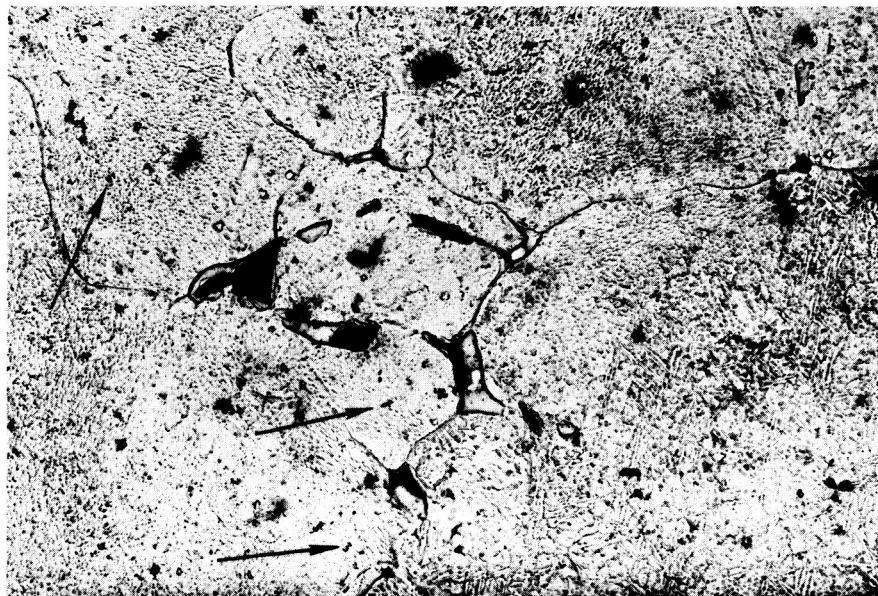
The Ti-6Al-4V alloy was cathodically charged at both 80 ma/in<sup>2</sup> and 0.4 amps/in<sup>2</sup>. After charging at 80 ma/in<sup>2</sup> the tritium appeared to be evenly distributed throughout the alpha matrix, Figure 10. Only very rarely was the tritium observed in the beta phase. Silver filaments were never clustered indicating no clustering of tritium atoms.

After charging at 0.4 amps/in<sup>2</sup>, the tritium appeared to form clusters of tritium atoms as shown in Figure 11. Also silver filaments appeared in clusters indicating that tritium was in clusters. Furthermore, a large number of the filaments appeared at alpha-beta boundaries and at alpha-alpha boundaries. No filaments were ever observed in the beta phase.



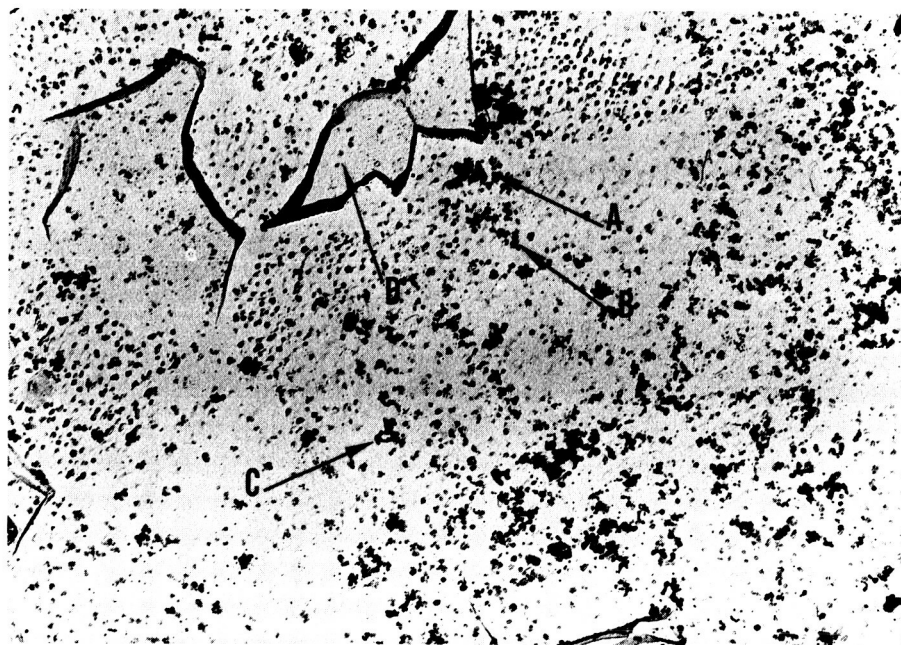
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Figure 8. Ti-5Al-2.5Sn After Cathodic Charging at  $80 \text{ mA/in}^2$  in  $4\% \text{ H}_2\text{SO}_4$  Made With Tritiated Water. Mag. 1050X.  
Arrow A points to hydride observed within grains.  
Arrow B points to hydride observed at grain boundaries.



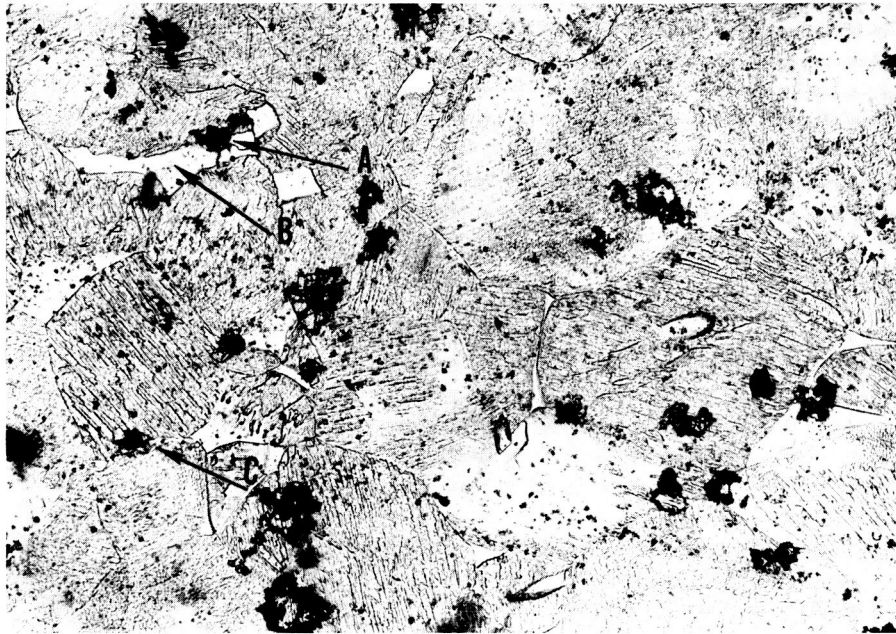
C-2508

Figure 9. Electron Microautoradiograph From Ti-5Al-2.5Sn Cathodically Charged at  $80 \text{ mA/in}^2$  For One Hour. Arrows point to typical silver filaments indicating position of tritium atoms. Mag. 10500X.



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Figure 10. Electron Microautoradiograph From Ti-6Al-4V Cathodically Charged at  $80 \text{ mA/in}^2$  For One Hour. Arrows A, B, and C point to typical silver filaments indicating position of tritium atoms. Arrow D points to beta phase.



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Figure 11. Electron Microautoradiograph From Ti-6Al-4V Cathodically Charged at  $0.4 \text{ amp/in}^2$  for One Hour. Silver filaments are in clusters indicating clustering of tritium. Arrow A points to cluster of silver filaments at an alpha-beta interface, Arrow B to the beta phase. Arrow C points to a cluster of silver filaments at an alpha-alpha boundary. Mag. 10500X.

#### 2.3.1.3 Ti-13V-11Cr-3Al Alloy

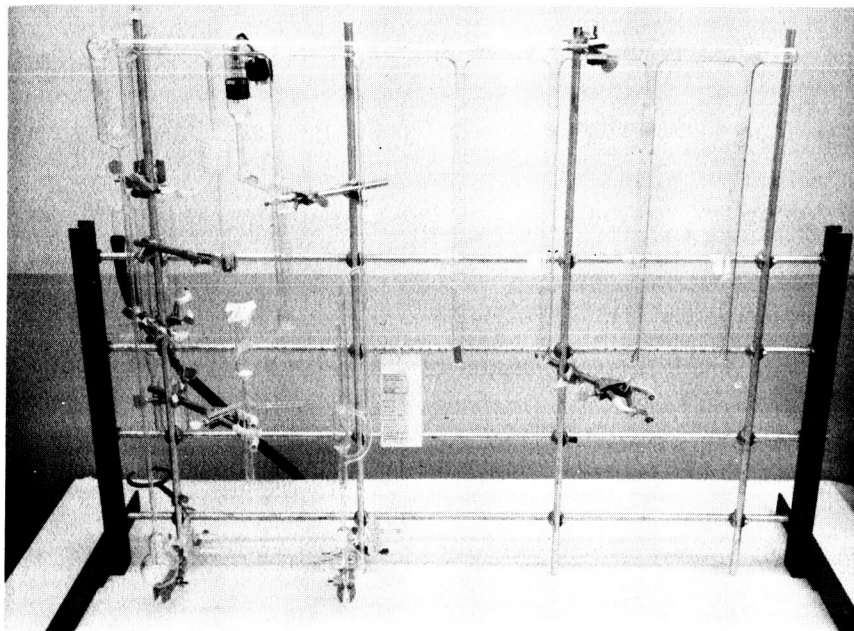
The Ti-13V-11Cr-3Al alloy was cathodically charged at 80 ma/in<sup>2</sup>. No silver filaments were observed indicating that cathodic charging of tritium was not successful.

The significance of these autoradiographs cannot be fully understood until the experiments described in the next section are completed.

#### 2.3.2 Gas Adsorption of Tritium in Titanium Alloys

Tritium gas was introduced into each of the four titanium alloys employing a modified Sieverts apparatus<sup>(5)</sup> designed and fabricated by Astropower Laboratory, as shown in Figure 12. The apparatus is comprised of a forepump, 2-stage mercury diffusion pump, ion gage, McLeod gage, tritium inlet system, and sample tubes. A mercury cut-off is used to isolate the vacuum manifold from the diffusion pump. The McLeod gage is accurately calibrated and covers the pressure range from 10<sup>-3</sup> to 1 torr. The ion gage was used only to determine the ultimate vacuum obtainable in the system during initial outgassing and pump down. The tritium inlet system employs a mercury-sealed fritted glass disc valve to admit the tritium to the vacuum manifold. Individual samples 1/4" x 1" were pickled in an aqueous solution of HNO<sub>3</sub>-HF and placed in vertical Vycor tubes which were attached to the Pyrex manifold using graded seals. With the manifold isolated from the vacuum pumps by the mercury cut-off, the total volume of the manifold was determined by expanding a known pressure of air (approximately 1 torr) from the known volume of the McLeod gage tube to the manifold. The pressure was remeasured - with the McLeod gage - and the system volume calculated from gas law.

In order to charge titanium alloys, the Vycor tubes containing an alloy sample was heated to 725°C ± 10°C, and held at this temperature until a final steady state pressure was attained in the entire system. The specimen tube was then cooled to ambient temperature and the final pressure was measured. The operation was repeated for each alloy until all the samples were charged. The amount of tritium introduced into each alloy is shown in Table V. The accuracy of the measurements by this technique is within 5%.



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Figure 12. Modified Sieverts Apparatus for Charging Tritium in Titanium Alloys

TABLE V  
CONCENTRATION OF TRITIUM IN TITANIUM ALLOYS

<u>Alloy</u>	<u>Concentration (ppm)</u>
Ti-5Al-2.5Sn	48
Ti-6Al-4V	41
Ti-8Al-1Mo-1V	43
Ti-13V-11Cr-3Al	43



The distribution of tritium in the titanium alloys will be determined by autoradiography during the next report period.

#### 2.4 Determination of Preferential Attack

In addition to determining the role of hydrogen in stress corrosion cracking of titanium alloys, the areas of local dissolution of initial sites will be determined by transmission electron microscopy. The experiment requires a preparation of thin foils of titanium alloys suitable for transmission electron microscopy, examining and photographing them, dipping the foils in corrosion solution (distilled water or 3% NaCl) for various lengths of time, and then re-examining in the electron microscope.

During this report period, the thickness of the sheet material has been reduced to 0.003 in. Ten specimens 1/2" x 1" of each alloy have been prepared. The thickness of original titanium alloy sheet was reduced to 0.010 in. by machining. Approximately 0.007 in. of material was removed by chem milling.

### 3.0 FUTURE WORK

A detailed analysis of the mechanism of stress corrosion cracking of the following alloys will be made:

1. Ti-5Al-2.5Sn
2. Ti-6Al-4V
3. Ti-8Al-1Mo-1V (beta discontinuous phase)
4. Ti-8Al-1Mo-1V (beta continuous phase)
5. Ti-13V-11Cr-3Al

Stress corrosion tests will be conducted in 3% NaCl and distilled water. Fracture time will be determined for each of the alloys as a function of load. The mode of crack propagation will be evaluated by electron fractography.

The distribution of tritium in various phases of titanium alloys will be studied by electron microautoradiography employing specimens which have been charged by gas absorption at 725°C and specimens which have been cathodically charged with tritium.

The sites of local dissolution of titanium alloy matrix will be evaluated by transmission electron microscopy. Thin foils of titanium alloys suitable for transmission of electron beam will be prepared. The foils will be examined and then dipped in distilled water or 3% NaCl and reexamined in the electron microscope. Preferential attack can be detected by increased transmission of the electron beam.

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